



# Dwell Time and Surface Parameter Effects on Removal of Silicone Oil From D6ac Steel Using TCA

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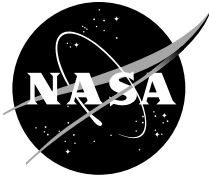
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## **TECHNICAL MEMORANDUM**

### **DWELL TIME AND SURFACE PARAMETER EFFECTS ON REMOVAL OF SILICONE OIL FROM D6AC STEEL USING TCA**

#### **1. INTRODUCTION**

Although silicone oil is considered to be one of the most difficult contaminants to remove from a surface, not much is known regarding factors that influence the ability to perform a successful cleaning operation. This study was conducted to evaluate the impact of dwell time, surface roughness, and surface activation state on 1,1,1-trichloroethane's (TCA's) effectiveness for removing silicone oil from D6ac steel. Silicone-contaminated test articles were washed with TCA solvent, and then the surfaces were analyzed for residue using Fourier transform infrared (FT-IR) analysis spectroscopy.

## 2. TEST DESCRIPTION

The test matrix is summarized in table 1. Surface roughness extremes from polished to grit blasted were evaluated. Silicone dwell times prior to TCA cleaning were 24 hr or 1 wk. The silicone was applied either when the surface was activated, meaning the oxidation growth was rapid due to recent grit blasting, or passivated, meaning the oxidation growth was minimal. Figures 1 and 2 show the test sample process flow. Control samples not contaminated with silicone but stored in a manner consistent with silicone-coated coupons were included.

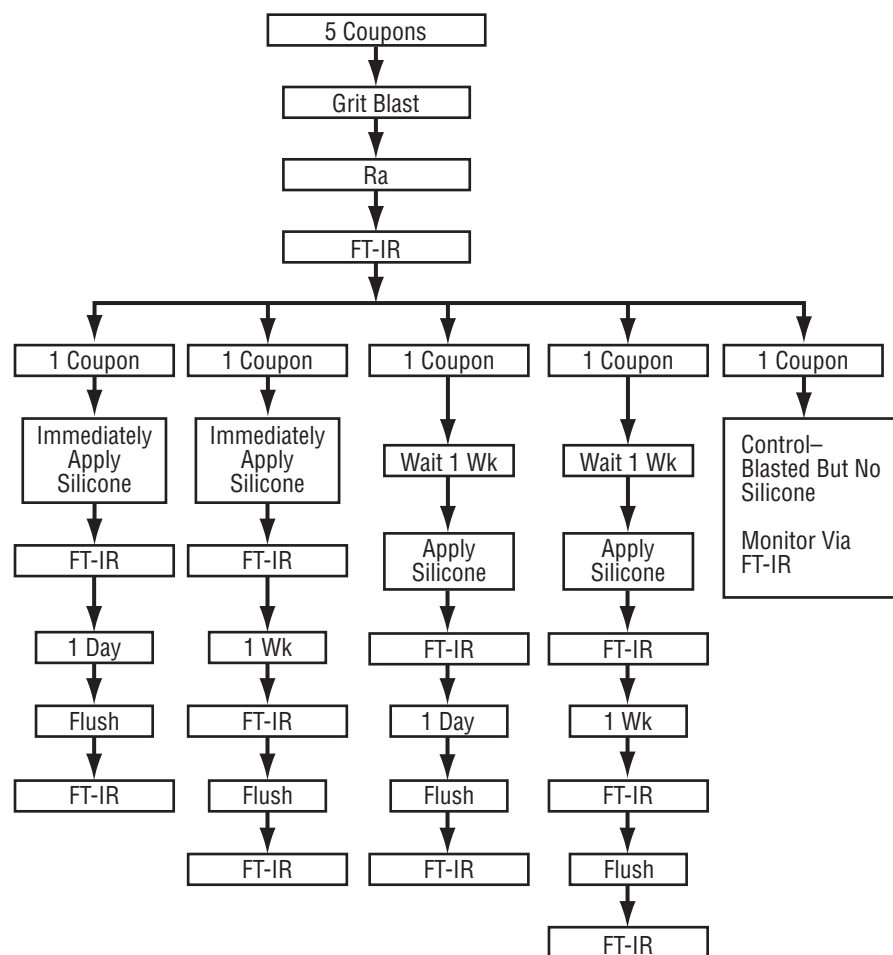
The dimensions of the D6ac steel test coupons were 2 by 3 by 0.25 in. The polished coupons were readied for use by processing through a surface grinder to achieve a smooth finish, and then by soaking in chloroform to remove the cutting oil. The rough coupons were prepared by soaking in chloroform and then grit blasting. Activated D6ac coupons were grit blasted  $\approx 1$  hr prior to the silicone application, while passivated coupons were grit blasted 1 wk prior to the silicone application.

The test articles were contaminated with 1,000 centistokes viscosity Dow Corning 200 silicone oil, using a Sono-Tek ultrasonic spray application system. The carrier solvent was TCA, and the coating levels ranged from 11 to 13  $\mu\text{g}/\text{cm}^2$ . The FT-IR analysis was performed using an FT-IR analysis spectrometer manufactured by Surface Optics Corporation, San Diego, CA—the SOC-400 spectrometer—fitted with a specular (polished coupons) or diffuse (grit-blasted coupons) reflectance accessory. Analysis parameters were 32 scans per spectrum at a resolution of 4  $\text{cm}^{-1}$ , noncontact mode, and five data points per coupon. The silicone methyl absorbance at  $\approx 1,260 \text{ cm}^{-1}$  was monitored. The estimated detection limits for silicone using these parameters were 0.2–0.3  $\mu\text{g}/\text{cm}^2$  for grit-blasted surfaces and 0.1–0.2  $\mu\text{g}/\text{cm}^2$  for polished surfaces.

Table 1. Test matrix.

Test No.	Surface Roughness		Silicone Dwell Time Prior to TCA Wash		Silicone Application Delay Time After Grit Blast	
	Polished	Grit Blasted	1 Day	1 Wk	1 Wk (Passivated)	Immediate* (Activated)
1	X		X			
2	X			X		
3		X	X		X	
4		X	X			X
5		X		X	X	
6		X		X		X

\*Within 1 hr after completion of grit blast

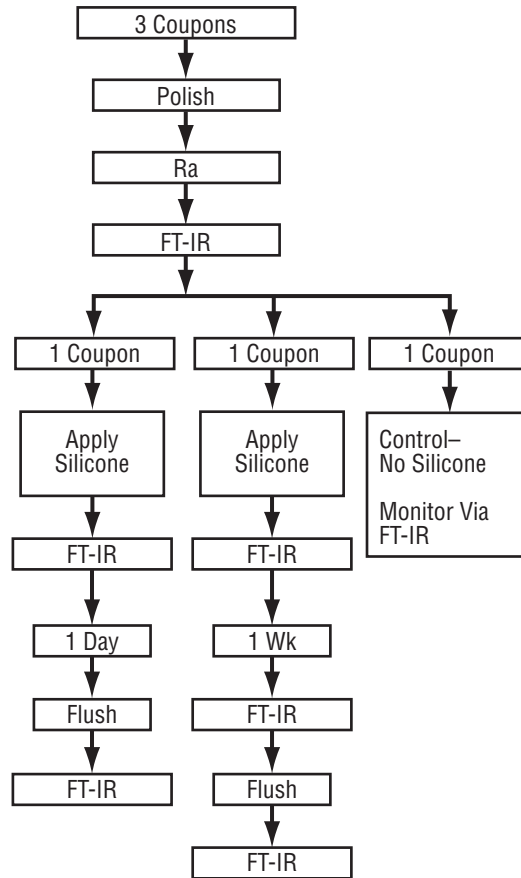


Immediately Apply Silicone (Within 1–2 hr, Target)

1 Day = 24 hr  $\pm$  6 hr

1 Wk = 7 Days  $\pm$  1 Day

Figure 1. Process flow for grit-blasted substrate.



Immediately Apply Silicone (Within 1–2 hr, Target)

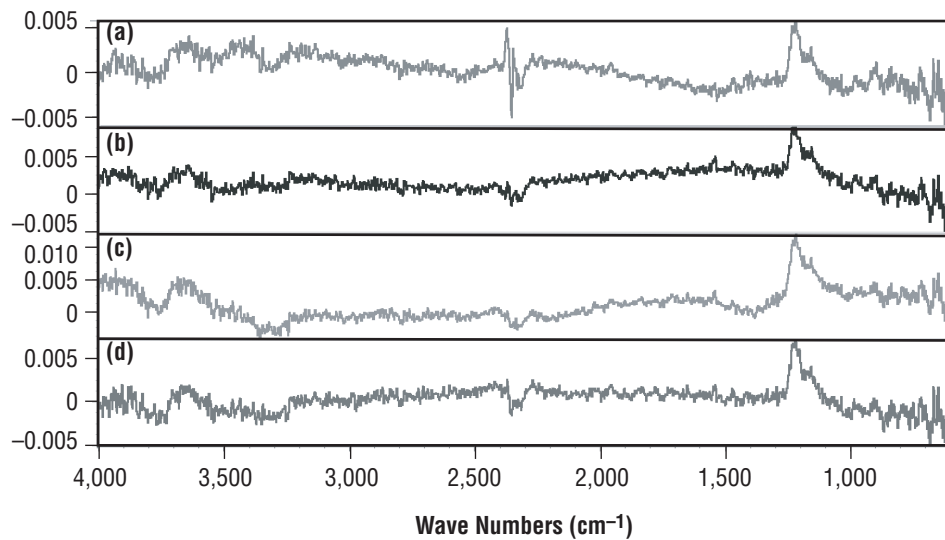
1 Day = 24 hr  $\pm$  6 hr

1 Wk = 7 Days  $\pm$  1 Day

Figure 2. Process flow for polished substrate.

### 3. TEST RESULTS

Figures 3 and 4 summarize FT-IR baseline analysis results for the test coupons. No contamination was detected prior to silicone application.

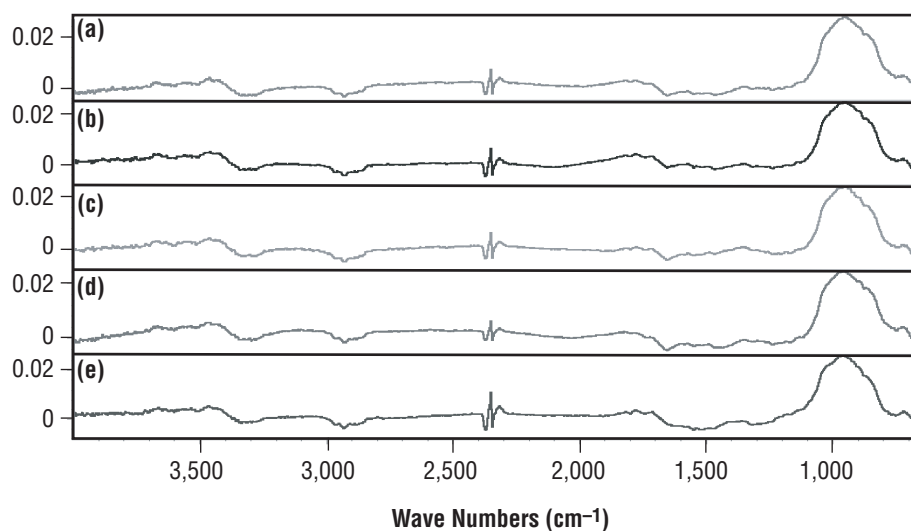


The absorbance @ 1,230 cm<sup>-1</sup> was due to oxide. The oxide was not expected to interfere with detection of the 1,260 cm<sup>-1</sup> absorbance of Dow 200 silicone. Baselines for the average of five spectra:

- (a) Coupon No. 1
- (b) Coupon No. 2
- (c) Coupon No. 3
- (d) Coupon No. 4

Figure 3. Analysis results of cleanliness verification of polished coupons prior to silicone application.





The absorbance @ 900  $\text{cm}^{-1}$  was due to oxide. The oxide was not expected to interfere with detection of the 1,260  $\text{cm}^{-1}$  absorbance of Dow 200 silicone. Baselines for average of five spectra:

- (a) Coupon No. 1
- (b) Coupon No. 2
- (c) Coupon No. 3
- (d) Coupon No. 4
- (e) Coupon No. 5

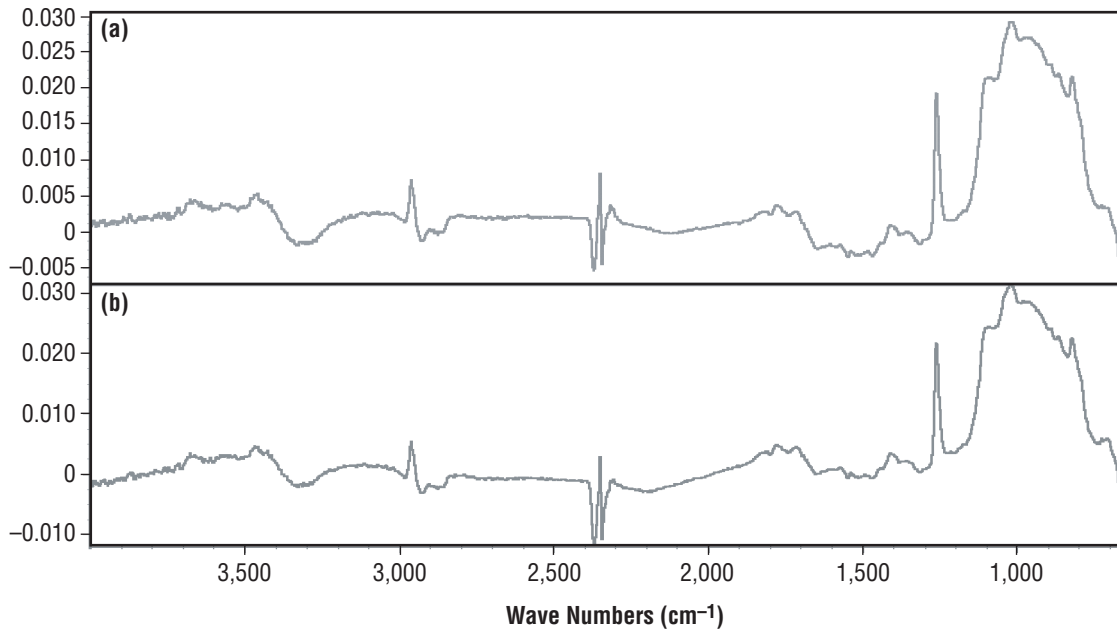
Figure 4. Analysis results of cleanliness verification of grit-blasted coupons immediately after blasting prior to silicone application.

Coupon surface roughness characteristics are summarized in table 2. Average surface roughness (Ra) in microinch values for the polished coupons ranged from 22 to 23  $\mu\text{in}$ , while average Ra values for the grit-blasted coupons ranged from 91 to 102  $\mu\text{in}$ .

Grit-blasted coupons Nos. 1 and 2 (activated) and polished coupons Nos. 1 and 2 were processed through the Sono-Tek ultrasonic spray application system, concurrently. The silicone coating level applied to these coupons was approximately 10–11  $\mu\text{g}/\text{cm}^2$  (figs. 5 and 6). The contaminant was visible to the unaided eye on the grit-blasted coupons and appeared as tiny “islands” on the surface. The oil was not easily observed on the polished test articles.

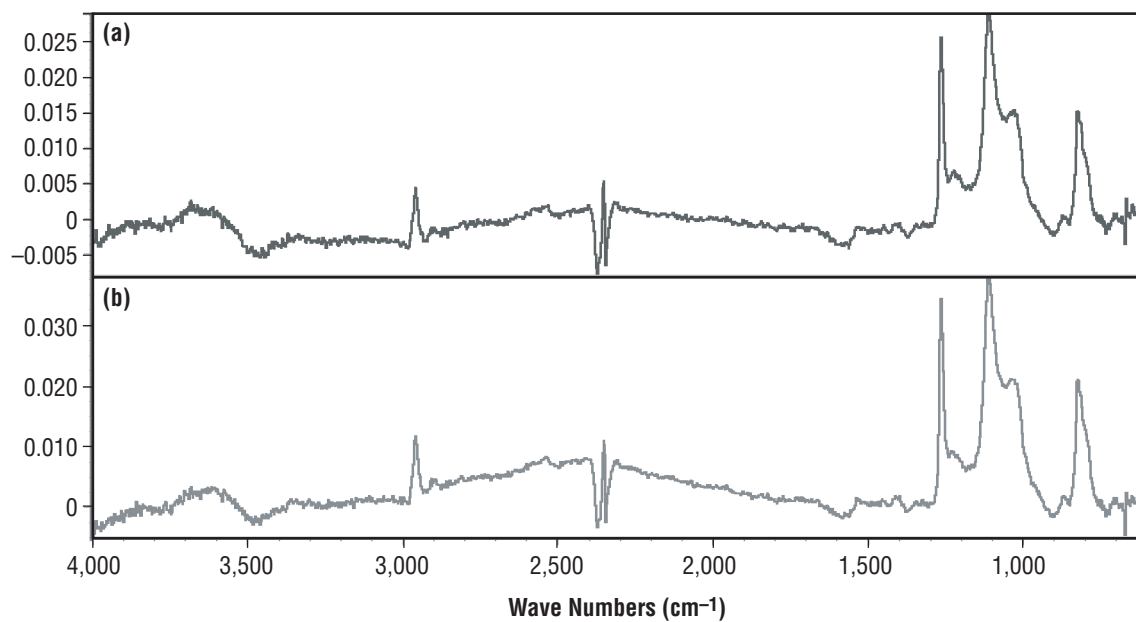
Table 2. Test article surface roughness.

Coupon	Ra ( $\mu\text{in}$ )					
	Measurement 1	Measurement 2	Measurement 3	Measurement 4	Measurement 5	Average
Polished No. 1	27	23	20	24	23	23
Polished No. 2	19	20	28	22	22	22
Polished No. 3	29	24	21	21	19	23
Polished No. 4	20	21	24	23	29	23
Blasted No. 1	97	98	97	89	112	99
Blasted No. 2	83	92	109	78	81	93
Blasted No. 3	92	105	86	87	84	91
Blasted No. 4	101	85	118	110	98	102
Blasted No. 5	110	96	83	98	101	98



Baselines for the average of five spectra:  
 (a) Coupon No. 1, 1,260 Ht = 0.0194 AU  
 (b) Coupon No. 2, 1,260 Ht = 0.0197 AU

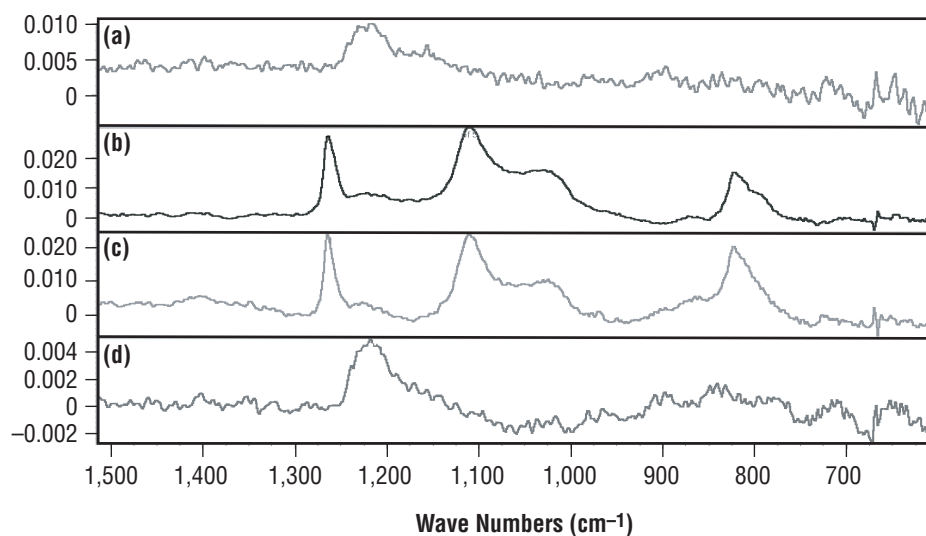
Figure 5. Analysis results of grit-blasted coupons Nos. 1 and 2 immediately following silicone application.



Baselines for the average of five spectra:  
(a) Coupon No. 1, 1,260 Ht = 0.0281 AU  
(b) Coupon No. 2, 1,260 Ht = 0.0336 AU

Figure 6. Analysis results of polished coupons Nos. 1 and 2 immediately following silicone application.

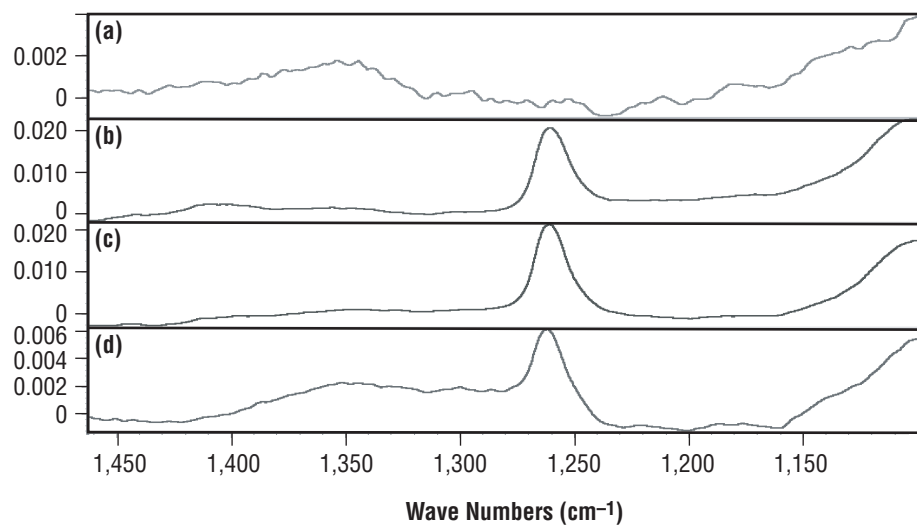
Grit-blasted coupon No. 1 and polished coupon No. 1 were reexamined with the FT-IR  $\approx 24$  hr after the silicone application and were found to be unchanged. The silicone “islands” on the grit-blasted sample had become slightly less distinct, but the average  $1,260\text{ cm}^{-1}$  magnitude was identical to zero time. Following FT-IR analysis, the two coupons were rinsed with TCA solvent in an attempt to remove the silicone. As shown in figure 7, the silicone was completely removed (below the detection limit of the FT-IR) from the polished coupon. As shown in figures 8 and 9, the silicone was not completely removed from the grit-blasted coupon, even after two flushing operations with TCA, a third operation where the coupon was rubbed with TCA-wetted Rymple cloth, and a final process where the TCA-wetted surface was scrubbed with a brush. The final silicone level for the grit-blasted coupon was  $\approx 1\text{ }\mu\text{g/cm}^2$ .



Baselines for the average of five spectra:

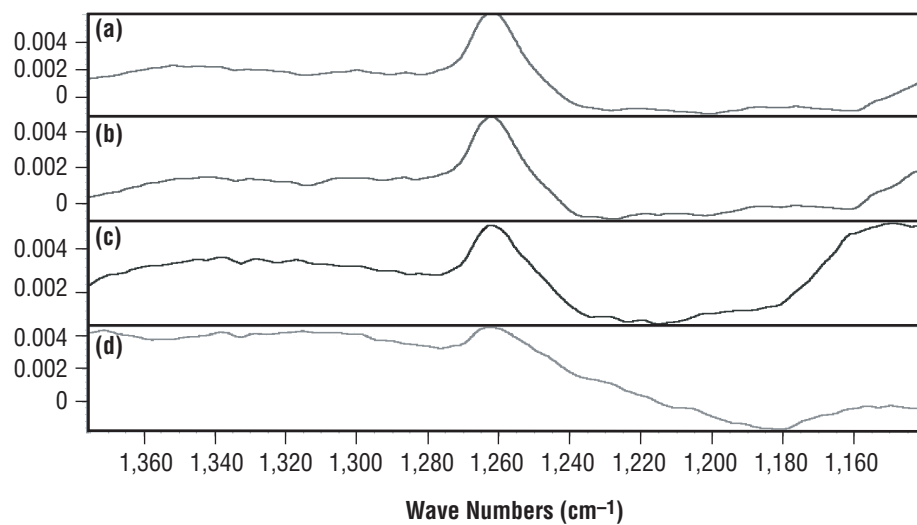
- (a) Immediately prior to silicone application
- (b) Immediately following silicone application ( $10\text{ mg/ft}^2$ )
- (c) 24-hr following silicone application and immediately prior to TCA flush
- (d) After initial attempt to remove  $10\text{ mg}$  of silicone by flushing with TCA; all four silicone absorbencies removed

Figure 7. Analysis results of complete silicone removal from polished coupon No. 1 after 24-hr staging.



Baselines for the average of five spectra:  
(a) Immediately after blasting and prior to silicone application  
(b) Immediately following silicone application  
(c) 24 hr after silicone application  
(d) Following initial TCA flush

Figure 8. Analysis results of incomplete silicone removal from grit-blasted coupon No. 1 after 24-hr staging.

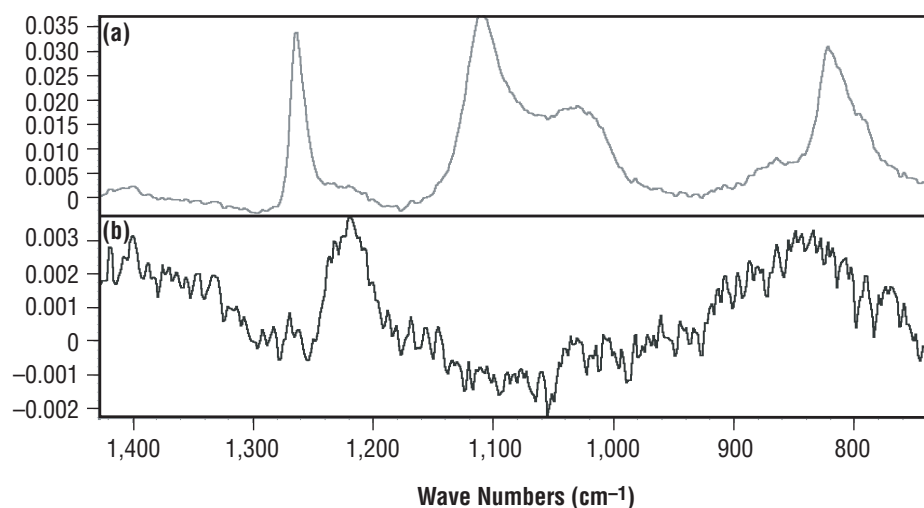


Baselines for the average of five spectra:

- (a) Following initial flush with TCA
- (b) Following second flush with TCA
- (c) Following two flushes with TCA and one wiping operation with TCA-wetted Rymple cloth
- (d) Following two flushes with TCA, one wiping operation with TCA-wetted Rymple cloth, and one scrubbing operation with TCA and a brush

Figure 9. Analysis results of grit-blasted coupon No. 1 following additional processing.

One week after silicone had been applied, polished coupon No. 2 and grit-blasted coupon No. 2 (activated) were reanalyzed using FT-IR to determine if the  $1,260\text{ cm}^{-1}$  absorbance intensity had changed relative to the initial inspection. Although the average  $1,260\text{-cm}^{-1}$  absorbance intensity remained unchanged for both test articles, the silicone on the grit-blasted coupon had spread to almost a uniform level across the surface. Figure 10 shows that the silicone was completely removed (below the detection limit of the FT-IR) from the polished coupon by rinsing with TCA. Figure 11 shows that the oil could not be completely removed from the grit-blasted coupon, even when the surface was rubbed with TCA-wetted Rymple cloth. The level of silicone remaining on the grit-blasted coupon was estimated at  $1.5\text{--}2.5\text{ }\mu\text{g}/\text{cm}^2$ .

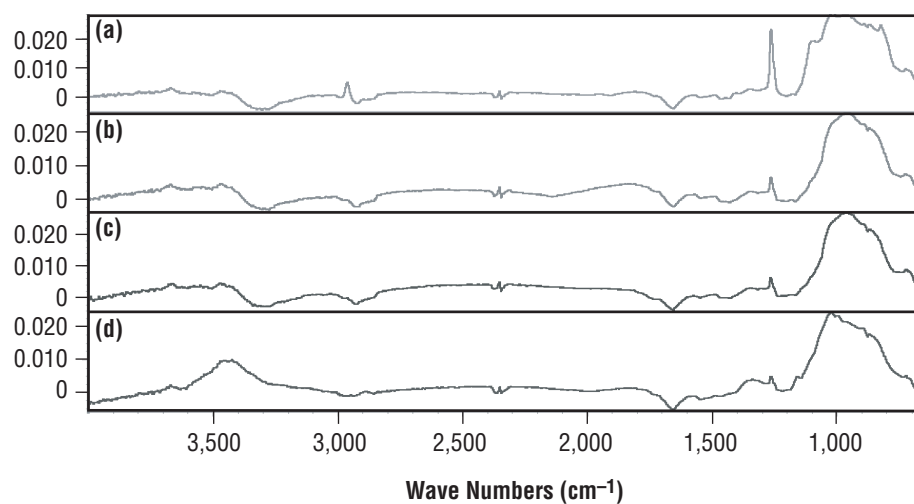


Baselines for the average of five spectra:

(a) Immediately prior to TCA flush

(b) Following flush with TCA; all four silicone absorbencies removed

Figure 10. Analysis results of complete silicone removal from polished coupon No. 2 after 1-wk staging.



Baselines for the average of five spectra:

(a) Immediately prior to initial TCA rinse

(b) After initial TCA rinse

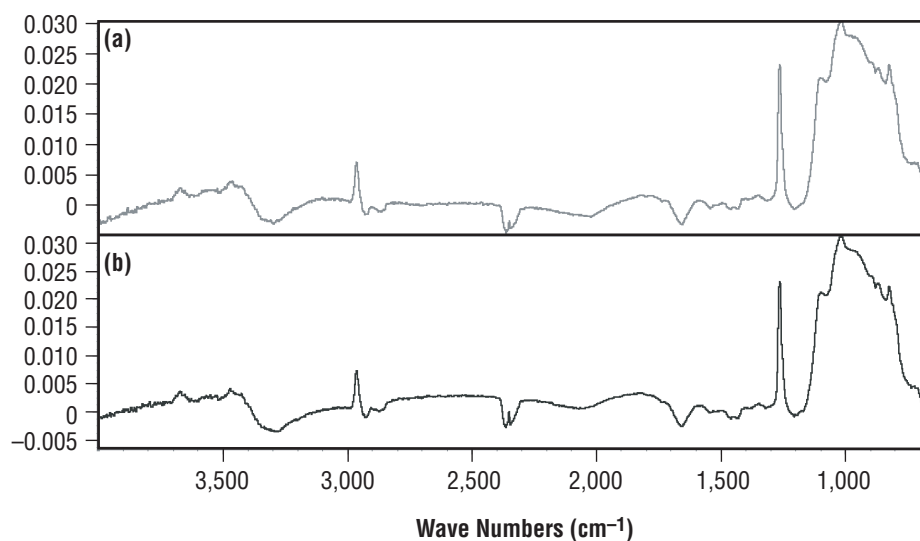
(c) Following second rinse with TCA

(d) Following second rinse with TCA, then one rubbing with TCA-wetted Rympie cloth

Figure 11. Analysis results of incomplete silicone removal from grit-blasted coupon No. 2 after 1-wk staging.



Grit-blasted coupons Nos. 3 and 4, which were allowed to oxidize for 1 wk, were contaminated with  $\approx 13 \mu\text{g}/\text{cm}^2$  of silicone (fig. 12).



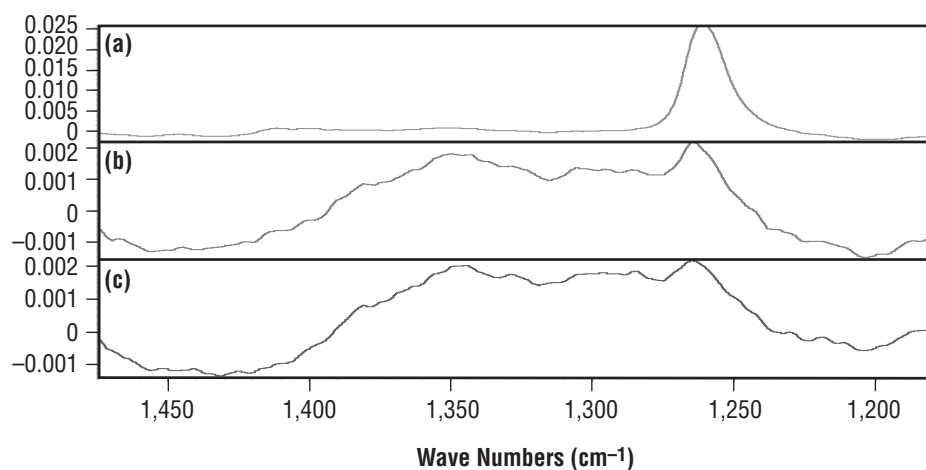
Baselines for the average of five spectra:

(a) Coupon No. 3,  $1,260 \text{ cm}^{-1} = 0.024 \text{ AU}$

(b) Coupon No. 4,  $1,260 \text{ cm}^{-1} = 0.0237 \text{ AU}$

Figure 12. Analysis results of grit-blasted coupons Nos. 3 and 4 immediately following silicone application.

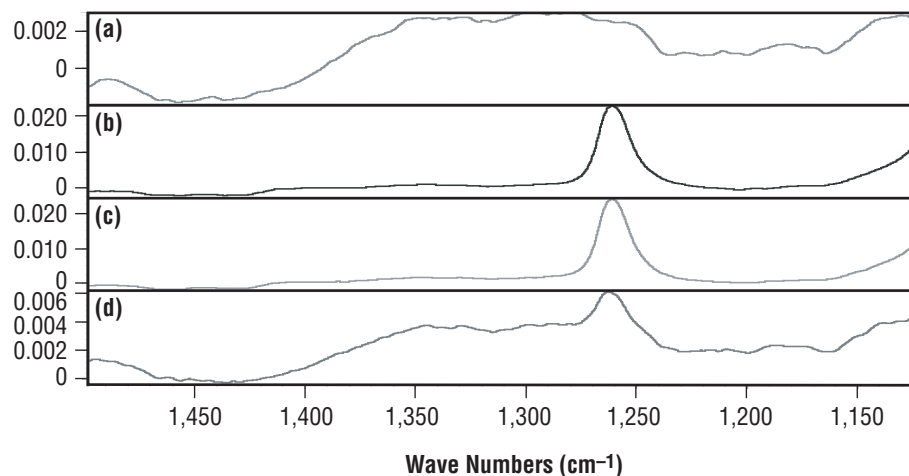
Coupon No. 3 was reanalyzed with FT-IR after 24 hr and was found to be unchanged with respect to the average  $1,260\text{ cm}^{-1}$  absorbance intensity. As illustrated in figure 13, attempts to remove silicone from this coupon using two TCA flushes were unsuccessful. The quantity of silicone remaining on the coupon was estimated to be  $0.5\text{--}1\text{ }\mu\text{g}/\text{cm}^2$ .



Baselines for the average of five spectra:  
(a) 24 hr after silicone application  
(b) Following initial flush with TCA  
(c) Following second flush with TCA

Figure 13. Analysis results of incomplete silicone removal from grit-blasted coupon No. 3 after 24-hr staging.

Coupon No. 4 was reanalyzed with FT-IR after 1 wk and was found to be unchanged with respect to the average  $1,260\text{ cm}^{-1}$  absorbance intensity. As illustrated in figure 14, attempts to remove silicone from this coupon using two TCA flushes were also unsuccessful. The quantity of silicone remaining on the coupon was estimated to be  $1\text{ }\mu\text{g}/\text{cm}^2$ .



Baselines for the average of five spectra:  
(a) Immediately prior to silicone application  
(b) Immediately after silicone application  
(c) 1 wk after silicone application  
(d) Following two flushing operations with TCA

Figure 14. Analysis results of incomplete silicone removal from grit-blasted coupon No. 4 after 1-wk staging.

#### **4. CONCLUSIONS**

The predominant factor affecting the ability to remove the silicone oil from D6ac steel by rinsing with TCA was surface roughness. Even with scrubbing, the oil could not be completely removed from grit-blasted test articles. The silicone was successfully washed from polished test articles after dwell times of up to 1 wk. The surface activation state did not appear to be important.

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